IS 6226-1 (1994): Recommendations for apparatus for chemicals analysis of metals,Part 1 Apparatus for determination of carbon by direct combustion [MTD 4: Wrought Steel Products]

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IS 6226 (Part 1) : 1994
(Reaffirmed 1999)

भारतीय मानक

धातु के रासायनिक विश्लेषण के लिए उपकरणों की सिफारिशें

भाग 1 प्रत्यक्ष दहन द्वारा कार्बन ज्ञात करने के उपकरण

(पहला पुनरीक्षण)

Indian Standard

RECOMMENDATIONS OF APPARATUS FOR CHEMICAL ANALYSIS OF METALS

PART 1 APPARATUS FOR DETERMINATION OF CARBON BY DIRECT COMBUSTION

(First Revision)

Second Reprint JANUARY 2005
(Including Amendment No 1)

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

February 1994

Price Group 2

Methods of Chemical Analysis of Ferrous Metals Sectional Committee, MTD 2

FOREWORD

This Indian Standard (Part 1) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 6226 (Part 1) was first published in 1971. The committee decided to revise this standard to make it more comprehensive by incorporating the following changes:

i) The purity limit of oxygen used for combustion of carbon has been increased from 99·0 to 99·5 percent.

ii) Legends have been given in Fig. 1 and Fig. 2.

Part 2 of this standard describes the apparatus for determination of sulphur by direct combustion.

AMENDMENT NO. 1 MARCH 1998
TO
IS 6226 (PART 1) : 1994 RECOMMENDATIONS OF APPARATUS FOR CHEMICAL ANALYSIS OF METALS

PART 1 APPARATUS FOR DETERMINATION OF CARBON BY DIRECT COMBUSTION

(First Revision)

(*Page* 3, *Fig.* 2) — Substitute the figure on page 2 for the existing:

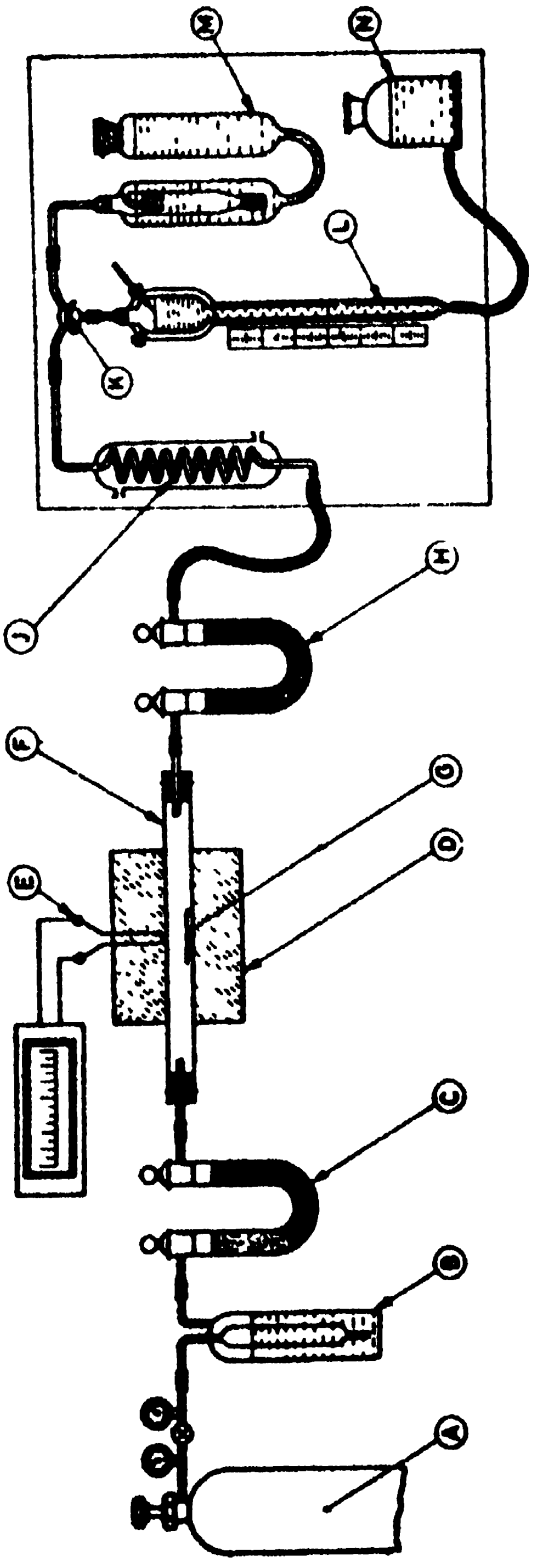


FIG. 2 APPARATUS FOR DETERMINATION OF CARBON BY VOLUMETRIC METHOD

(MTD 2)

Reprography Unit, BIS, New Delhi, India

Indian Standard

RECOMMENDATIONS OF APPARATUS FOR CHEMICAL ANALYSIS OF METALS

PART 1 APPARATUS FOR DETERMINATION OF CARBON BY DIRECT COMBUSTION

(First Revision)

1 SCOPE

This standard (Part 1) recommends apparatus used for the determination of total carbon in metals ores and minerals by gravimetric and volumetric methods.

2 APPARATUS FOR DETERMINATION OF CARBON BY THE GRAVIMETRIC METHOD

The apparatus consists of three parts: (a) oxygen cylinder and purifier; (b) a furnace with combustion tube; and (c) the train for purifying and absorbing the carbon dioxide evolved by combustion of the carbon present in the sample. The three parts, which are connected to one another by tubes and hermetically sealed with stoppers, are shown in Fig 1.

2.1 First Part

2.1.1 *Source of Oxygen*

An oxygen cylinder *A* containing oxygen of at least 99·5 percent purity, free from organic contaminants and provided with a two stage reduction valve and a mercury valve *B* to facilitate even and adequate flow of oxygen.

2.1.2 *Drying and Purifying Unit for Oxygen*

Consists of a U-shaped tube *C* (diameter 25 mm and height 100 mm approximately), containing soda asbestos and anhydrous magnesium perchlorate separated by glass wool.

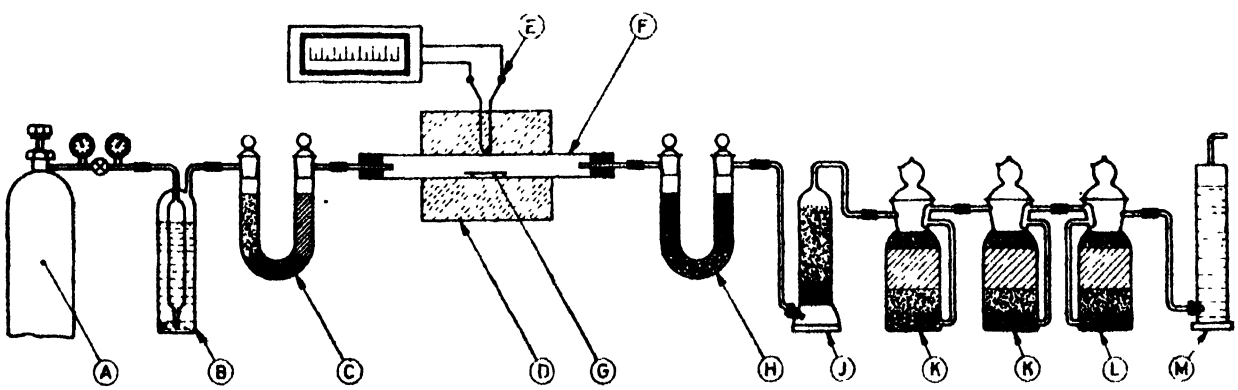
2.2 Second Part

2.2.1 *Furnace*

Wire-wound or resistor rod furnace *D* capable of attaining a maximum temperature of 1 200°C with a heating zone of about 100 mm in central position is used. Alternatively and for higher temperatures, induction furnaces can be used.

2.2.2 *Thermocouple*

For measuring the temperature, thermocouple *E* is used. The tip of the thermocouple, protected by a sheath, is placed near the external surface of the combustion tube. The relation between the internal tube temperature and the pyrometer reading should be established.



A — Oxygen cylinder
B — Mercury valve
C — U-shaped tube
D — Wire wound or resistor rod furnace
E — Thermocouple
F — Combustion tube
G — Boat or crucible
H — U-shaped tube
J — Drying bulb
K — Absorption bulb
L — Absorption bulb
M — Flow meter

FIG. 1 APPARATUS FOR DETERMINATION OF CARBON BY GRAVIMETRIC METHOD

2.2.3 *Combustion Tube*

Combustion tube *F* made of refractory material, such as porcelain, sillimanite, quartz, clay which are not porous at the test temperature, may be used. The tube should be about 650 mm long with an inside diameter 20 to 30 mm.

2.2.4 *Boat/Crucibles*

The boat *G* (length 75 to 90 mm, width 12 to 14 mm and depth 8 to 9 mm) or crucibles may be of refractory materials, such as fireclay, porcelain, zirconia, etc. Before use, the crucible should be heated at 1 100°C in oxygen or air until a constant blank is obtained.

2.3 Third Part

2.3.1 *Carbon Dioxide Purifying Unit*

It consists of a U-shaped tube *H* containing manganese dioxide granules or platinized silica-gel heated to about 400°C, plugged with thick asbestos wool on either side and a drying bulb *J* containing anhydrous magnesium perchlorate. This unit removes finely-divided solid metallic oxides, oxides of sulphur and selenium and dries the gases before they enter the weighed absorber.

2.3.1.1 Additional components in the purification train may be required in some cases. For complete conversion of CO to CO_2, especially while using in induction heating, a heated oxidation catalyst tube containing CuO, is necessary. A second tube containing manganese dioxide may be used when the sulphur content of the sample is very high. Chlorides may also be removed by manganese dioxide, or by pumice impregnated with a mixture of sodium thiosulphate and potassium iodide or 20 to 30 mesh zinc metal pre-heated to 300°C to 325°C. Other materials may be substituted for those listed; for example, potassium permanganate solution may replace the manganese dioxide and sulphuric acid may be used for removing water vapour, provided satisfactory results are obtained. The materials used in the purification train shall be checked frequently to ensure that their absorbing capacity has not been exhausted.

2.3.2 *Absorption Train*

Two absorption bulbs *K* charged with absorbent for carbon dioxide are used. The most desirable absorbent for carbon dioxide is 20 to 30 mesh inert base ascarite, impregnated with sodium hydroxide followed by anhydrous magnesium perchlorate at the exist end. The latter absorbs the water formed during the absorption reaction. A layer of glass wool placed at the bottom and top of the bulbs. The bulbs *K* should not weigh over 100 g. They should always be weighed filled with oxygen and against a like counter poise.

2.3.3 An unweighed absorption bulb *L* charged with absorbents and glass wool in the same way as bulbs *K* but facing the opposite way to the latter is used to trap any carbon dioxide or moisture from the atmosphere. The flow of oxygen through the train is regulated to the desired value as indicated by the flow meter *M*.

3 APPARATUS FOR DETERMINATION OF CARBON BY THE VOLUMETRIC METHOD

The apparatus consists of three parts: (a) oxygen cylinder and purifier; (b) a furnace with combustion tube; and (c) the train for purifying and absorbing the carbon dioxide from the combustion of the carbon present in the sample. The three parts, which are connected with one another by tubes and hermetically sealed with stoppers, are shown in Fig. 2.

3.1 For the description of first two parts, *see* **2.1** and **2.2**.

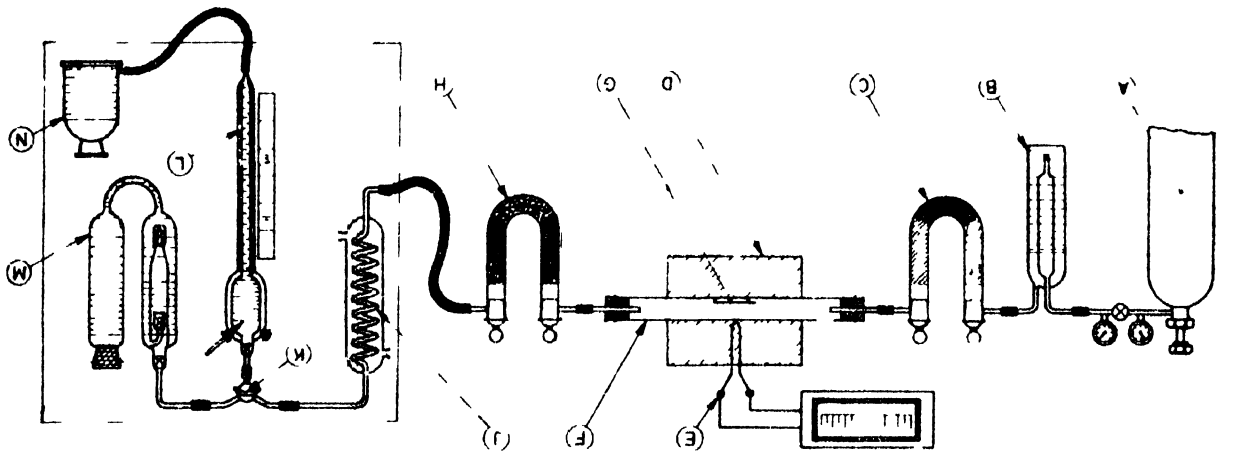
3.2 Third Part

3.2 1 *Train for Purifying and Absorbing Carbon Dioxide*

It consists of a U-shaped tube *H* containing manganese dioxide granules plugged with thick asbestos wool on either side to trap any sulphur combustion products and dust carried over by the stream of gas, a water cooled capillary coil *J* (inside dia 1·5 mm) one end of which is connected to the U-shaped tube *H* and the other end through a 3-way stop-cock *K* to the specially calibrated burette *L* cooled by water jacket; the absorption receiver *M* with liquid trap, containing solution of potassium hydroxide (500 g per litre), used for absorbing the carbon dioxide and connected to the combustion tube through tap *K* and the purifying train.

3.2.2 The bottom of the burette is connected by a rubber tube to a levelling bottle *N* containing 10 percent sodium chloride solution acidulated with sulphuric acid and coloured red with methyl orange. There is a scale which can be slided along the burette itself.

3.2.3 The capacity of the specially calibrated burette should be 400 ml for samples weighing 1 g with carbon content up to 1·0 percent and about 600 ml for samples weighing 1 g with carbon content up to 4·5 percent.



A — Oxygen cylinder
B — Mercury valve
C — U-shaped tube
D — Wire wound or resistor rod furnace
E — Thermocouple
F — Combustion tube
G — Boat or crucible
H — U-shaped tube
J — Water cooled capillary coil
K — Three way stop cock
L — Calibrated burette
M — Absorption receiver
N — Levelling bottle

FIG. 2 APPARATUS FOR DETERMINATION OF CARBON BY VOLUMETRIC METHOD

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This Indian Standard has been developed from Doc : No. MTD 2 (3784).

Amendments Issued Since Publication

Amendment No.	Date of Issue	Text Affected

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